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Stability Evaluation of Ambient Concentrations of Sulfur Dioxide, Nitric Oxide, and Nitrogen Dioxide Contained in Compressed Gas Cylinders

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Compressed gas samples of sub-part per million concentrations of sulfur dioxide, nitric oxide, and nitrogen dioxide were evaluated for long and short term stability. Except for several stainless steel tanks, the samples were contained in aluminum cylinders. A degree of stability was achieved over the short term, however, all of the samples were considered unstable over the term of the evaluations. Utilization of ambient level compressed gas samples may be considered where relative stability is required for no more than two or three months.

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STABILITY EVALUATION OF AMBIENT CONCENTRATIONS OF SULFUR DIOXIDE, NITRIC OXIDE, AND NITROGEN DIOXIDE CONTAINED IN COMPRESSED GAS CYLINDERS

by

Berne I. Bennett
Quality Assurance Branch
Environmental Monitoring and Support Laboratory
Research Triangle Park, North Carolina 27711

ENVIRONMENTAL MONITORING AND SUPPORT LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
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FOREWORD

The assessment of air quality must rely on instrumental measurements of specific air contaminants. Air monitors require a calibration source that is often contained in a compressed gas cylinder. The Environmental Monitoring and Support Laboratory continues to investigate the commercial availability of stable reactive gas standards.

This report presents the results of tests that were conducted over a 2-yr period to evaluate the trustworthiness of available sub-ppm standards of sulfur dioxide, nitric oxide, and nitrogen dioxide contained in aluminum compressed gas cylinders.

Thomas R. Hauser, Director
Environmental Monitoring and Support
Laboratory
Office of Research and Development

ABSTRACT

Compressed gas samples of sub-ppm concentrations of sulfur dioxide, nitric oxide, and nitrogen dioxide were evaluated for long- and short-term stability. Except for several stainless steel tanks, the samples were contained in aluminum cylinders. A degree of stability was achieved over the short term; however, all of the samples were considered unstable over the term of the evaluations. Utilization of ambient level compressed gas samples may be considered where relative stability is required for no more than 2 or 3 months.

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LIST OF ABBREVIATIONS AND SYMBOLS

ABBREVIATIONS

approx.	– approximately
°C	– degree Celsius
cm	– centimeter
EMSL	– Environmental Monitoring and Support Laboratory
EPA	– U.S. Environmental Protection Agency
m^3	– cubic meter
min	– minute
mV	– millivolt
NBS	– National Bureau of Standards
nm	– nanometer
o.d.	– outside diameter
ppm	– part per million
QAB	– Quality Assurance Branch
RTP	– Research Triangle Park, North Carolina
yr	– year

SYMBOLS

CO	– carbon monoxide
NO	– nitric oxide
NO_2	– nitrogen dioxide
O_3	– ozone
SO_2	– sulfur dioxide
R^2	– coefficient of regression squared

ACKNOWLEDGMENT

Recognition is due staff members of the Ambient Air Section, Quality Assurance Branch, and of Northrop Services, Inc., for diligent performance of the many analyses contributing to development of this report.

SECTION 1

INTRODUCTION

The Quality Assurance Branch (QAB), Environmental Monitoring and Support Laboratory (EMSL), U.S. Environmental Protection Agency at Research Triangle Park, North Carolina (EPA-RTP) has been engaged in evaluating the stability of several reactive gases contained in compressed gas cylinders. The stable containment of sub-ppm concentrations of sulfur dioxide (SO_2), nitric oxide (NO), and nitrogen dioxide (NO_2) was not considered possible until 1973, when experiments suggested that stability might be achieved by use of high pressure aluminum cylinders (1). The achievement and preservation of stability of low concentration levels of reactive mixtures require the application of a series of cylinder pretreatments and subsequent scrupulous avoidance of atmospheric contamination.

Several sizes of aluminum cylinders were available at the onset of this study. Since the size 30 aluminum cylinders (0.84 m^3 of gas) were being used successfully in the EPA carbon monoxide (CO) performance surveys, little consideration was initially given to any other size. Experiments demonstrated, however, that size 30 cylinders contained insufficient gas for long-term use and were frequently depleted before completion of the study. Cylinders subsequently obtained for the study were of the larger size 80 (2.24 m^3 of gas), which proved to contain ample volume. Also utilized were several cylinders of sizes 150 (4.2 m^3 of gas) and 100 (2.8 m^3 of gas).

Twenty-three samples of SO_2 in a concentration range of from 0.1 to 0.9 ppm were evaluated over a 26-month period. The material appraised was prepared in five different lots, including two stainless steel samples.

Twenty-five aluminum cylinders and one of stainless steel comprised the sample material for the NO evaluation. The concentration range examined was from 0.2 to 1.1 ppm.

Twenty-six samples of NO_2 in a concentration range of from 0.05 to 0.25 ppm were evaluated over a 10-month period; this evaluation involved aluminum cylinders only.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

SO_2 samples acquired early in the evaluation program were both inaccurate and unstable. Both of these qualities improved in the succeeding groups of test samples, but none of the SO_2 cylinders achieved absolute stability. Several exhibited minimal decreases over a period of 1 to 3 months; over the term of the evaluation, progressively greater reductions occurred. Careful avoidance of atmospheric contamination was necessary to prevent any residual moisture from entering the cylinders by back diffusion. The possibility of operator contamination of the samples could not be excluded with absolute assurance; however, all analysts handling the cylinders were cognizant of the importance of preventing vitiation of the samples.

Eight of the 11 cylinders listed in Table 6 exhibited a constant or nearly unchanged NO concentration for 9 months, demonstrating that it is possible to contain sub-ppm concentrations of NO in aluminum cylinders. Between the test periods of May and December 1976 (Table 5), 11 of the 14 cylinders remained unchanged. The test period between November 1977 and February 1978 was one of relative stabilization. Despite such periods of stability, however, the overall performance during the assessment was one of unreliable stability. The degree of short-term stability may be considered sufficient where relative consistency over a 2- or 3-month period is adequate.

Although concentrations of several of the NO_2 cylinders remained unchanged for 3 months, the magnitude of the decreases of many others over the survey period was so great that their utilization would be of questionable value.

Even though absolute stability was not attained in our tests, the achievement of brief periods of relative stability suggests that advances in cylinder treatment and gas handling will, in time, enable the production of stable and reliable ultralow concentrations of reactive gas mixtures.

The evaluation of low level reactive compressed gas samples is a continuing effort. Because of the need for reliable sub-ppm standards, samples from as many different gas suppliers as practicable will be examined in a future stability study.

SECTION 3

EXPERIMENTAL

SULFUR DIOXIDE

Principle of Measurement

All measurements were obtained with Bendix® Model 8300 Total Sulfur Analyzers (2) employing the principle of photometric detection of luminescence resulting from the burning of sulfur compounds in a hydrogen-rich flame. The primary components in this analytical process are the detector cell, burner chamber, photomultiplier assembly, and gas and sample conduction tubing. Tubing to the burner chamber that comes into contact with the sample is constructed of Teflon®. The burning sulfur produces luminescence in the 394-nm wavelength region and is coupled through an optical filter to the photomultiplier tube. The photomultiplier converts the luminescence into an electrical signal that is proportional to the amount of sulfur burned. The output from the detector cell is linearized by an exponential amplifier and presented as a continuous readout on the panel meter and a continuous signal to a 10-mV recorder. A temperature controller maintains the burner block temperature at 110°C to stabilize the measurement process.

Calibration

Dynamic Dilution—

One method of calibration was dynamic dilution of a high concentration of National Bureau of Standards (NBS) certified reference standard with SO_2 free carrier air to produce working standards in the range of from 0.05 to 1.0 ppm. An Airco® Model MB 532 Gas Blender incorporating three calibrated flow

controllers was used to blend the SO_2 with the diluent gas purified by use of a Bendix® Model 8833 Air Purification System. The blended mixture was delivered through a glass mixing chamber to a Teflon® sampling manifold. Since the analyzer is sensitive to small pressure changes, the sample pressure was monitored by use of a water manometer and adjusted to 0.1 inches water by controlling the amount of sample gas vented. Teflon® tubing (0.25 inches o.d.) conducted the sample gas to the analyzer at a flow rate of approximately 175 c^3/min. Diagrams of the blending and delivery system are shown in Figures 1a and 1b. Five SO_2 concentrations and a zero were obtained in this manner. The method of least squares was used to achieve a linear calibration equation for recorder response v. SO_2 concentration.

Permeation Tube—

Calibrations were also performed using a Bendix® Model 8850 Permeation System with a 5-cm calibrated permeation tube. A diagram of this permeation oven is shown in Figure 2. An additional dilution step was added to obtain lower concentration standards, enabling a calibration range of from 0.08 to 0.70 ppm. A five point plus zero linear calibration equation was determined for recorder response v. concentration by the method of least squares. A comparison between the two calibration methods showed that agreement over the range tested was between 0.3% and 3%.

Analysis

Sample gas was conducted from the cylinder to the analyzer as shown in Figure 3. A low-volume Veriflo® Type 660 Pressure Regulator (without gauges) was utilized to reduce the chances of back contamination of the cylinders and to assure rapid and complete sample exchange. A pressure of 0.1 inches water was maintained in the sample line. The sample was permitted to flow until a fixed straight line trace was obtained on a strip chart recorder. The initial sample cylinder commonly required about 60 min for a stabilized trace. Subsequent samples needed approx. 15 min each to achieve a fixed response. The SO_2 concentrations proportional to the response were derived from the calibration equation. Duplicate analyses on different days were obtained in most

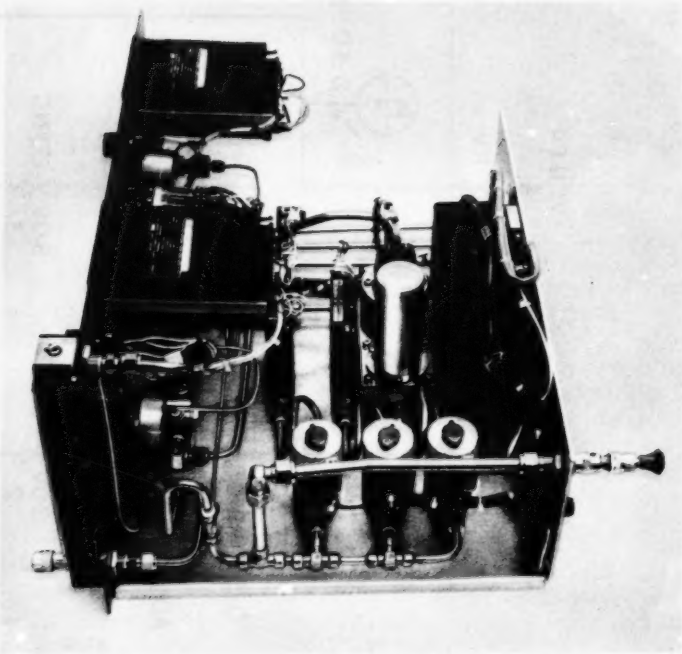

Figure 1a. Gas blending system.

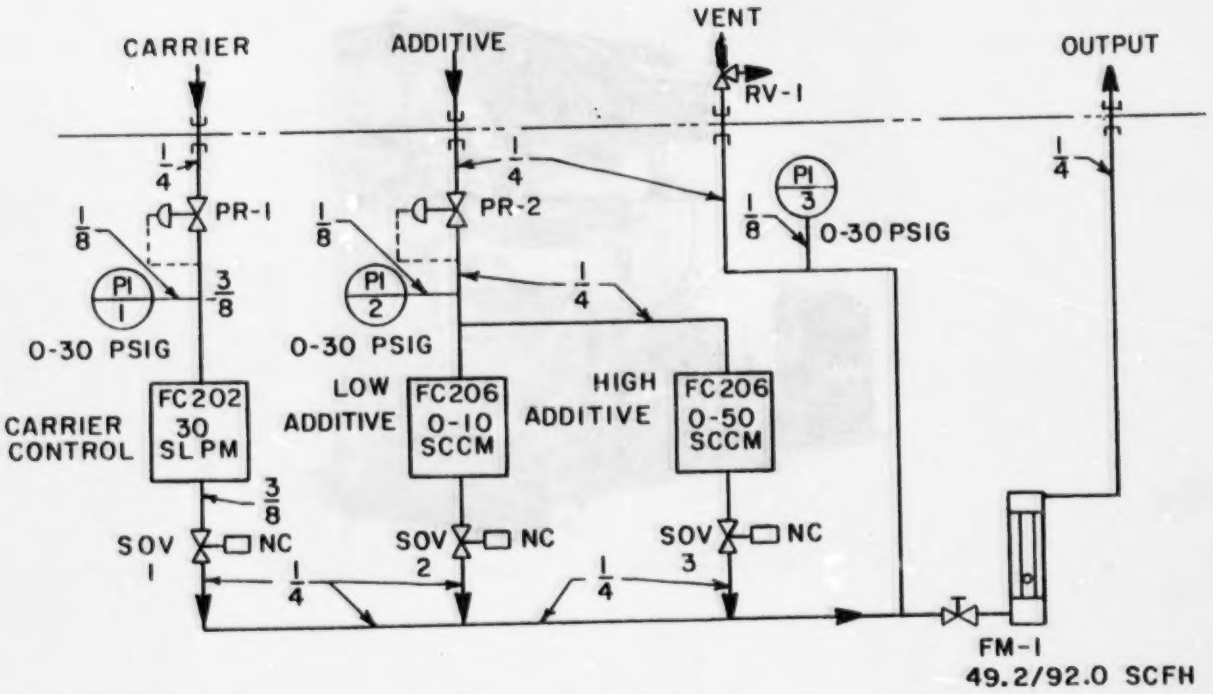

Figure 1b. Schematic of gas blending system.

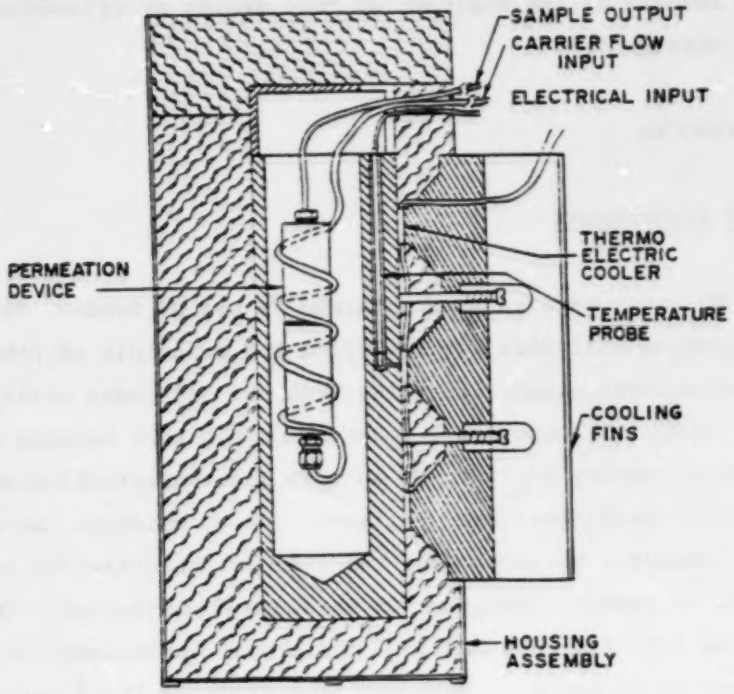

Figure 2. Permeation tube oven assembly.

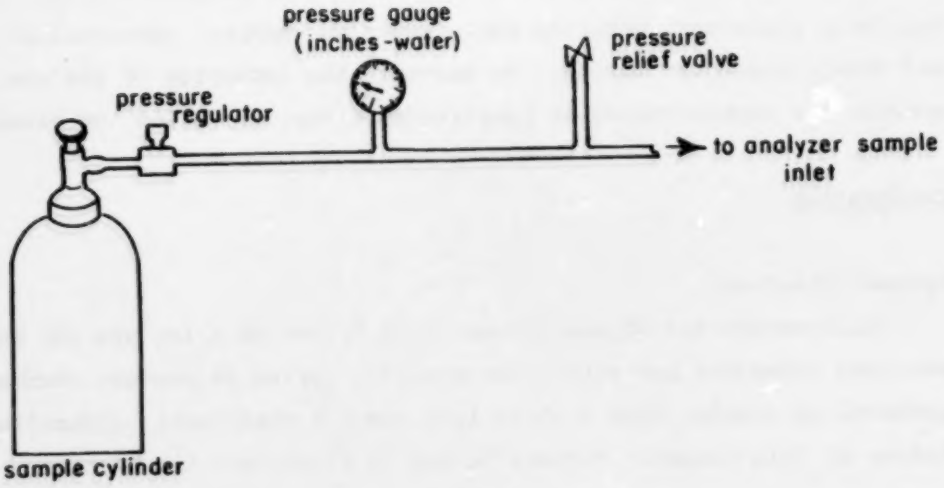

Figure 3. Delivery of sample gas to analyzer.

cases. The results of the analyses of four groups of cylinders are summarized in Tables 1 through 4.

OXIDES OF NITROGEN

Principle of Measurement

NO and NO_2 measurements were obtained by use of Bendix® Model 8101B Oxides of Nitrogen Analyzers (3) employing the principle of photometric detection of chemiluminescence resulting from the gas phase reaction of NO and ozone (O_3). Since this gas phase reaction only occurs between NO and O_3, it is necessary to convert NO_2 into NO so that its concentration can be measured. Two temperature controllers are utilized: one to maintain the block temperature of the catalytic converter and the other to maintain the reaction chamber block at 45°C to prevent formation of moisture condensation. The reaction chamber of the detector cell provides the proper environment to assure the gas phase reaction of NO and O_3. This reaction produces light particles or photons that pass through the optical filter to the photomultiplier tube where they are converted into electrical energy and amplified. The degree of gas phase reaction is directly proportional to the amount of NO present in the air sample, thus producing a proportional number of photons.

The principal components in this analytical process are the O_3 generator, catalytic converter, detector cell, reaction chamber, photomultiplier assembly, and sample conductor tubing. To maintain the integrity of the reactive component, the sample tubing is constructed either of Teflon® or glass.

Calibration

Dynamic Dilution—

Calibration for NO was accomplished by use of a 100 ppm NBS certified standard reference gas with a gas blending system to produce standard NO concentrations ranging from 0.15 to 1.20 ppm. A five point calibration was obtained by this method. Figures 1a and 1b illustrate the blender. A baffled

glass mixing bulb was installed at the mixture outlet to promote complete mixing. The mixture was conducted to the analyzer through Teflon® tubing (0.25 inches o.d.). Sample pressure was monitored and controlled in the manner described above (SO_2 Analysis section). The method of least squares was used to determine a linear calibration equation for analyzer response v. NO concentration. Coefficients of linearity (R^2) of 0.9999 were commonly obtained.

Permeation Tube—

For NO_2, calibration was achieved by use of Bendix® Model 8850 Permeation Systems and standard permeation tubes (NBS Standard Reference Material No. 1629). Five point calibrations ranging from 0.17 to 1.4 ppm were obtained. The diluent gas was high purity nitrogen which had passed through charcoal and Ascarite® adsorbents (4) to remove residual NO_2. The blended standards were delivered to the analyzer through Teflon® tubing (0.25 inches o.d.). Sample pressure was maintained at 0.1 inches water. A linear calibration equation for instrument response v. NO_2 concentration was determined by the method of least squares. Response over the working range was linear, with an R^2 of 0.9999.

Analysis

Inert Teflon® and stainless steel were required to convey sample gas from the cylinder to the analyzer. A low volume Veriflo® Type 660 pressure regulator (without gauges) was employed to reduce the risk of back diffusion and concomitant contamination of the cylinder. Use of this pressure regulator lessened the equilibration time required to achieve a fixed recorder trace from >1 hr to <10 min. Figure 3 illustrates delivery of sample gas to the analyzer. Positive pressure of 0.1 inches water was maintained. A strip chart recorder documented the analyzer response of individual evaluation cylinders. NO and NO_2 concentrations corresponding to recorder response were calculated from the calibration equations. To determine the precision of the measurements, duplicate analyses were performed on different days. The cumulative results for each cylinder are shown in Tables 5 through 7.

SECTION 4

RESULTS AND DISCUSSION

SULFUR DIOXIDE

In the early phase of the evaluations, some large discrepancies between the supplier's certifications and our determinations were encountered. In all such instances, the affected cylinders were tested by an independent laboratory whose corroborative tests (together with our own) indicated substantial errors in the supplier's certifications. The cylinders involved were those in Group 1 (Table 1). A progressive decline in SO_2 was characteristic of the entire group. In several samples cited in Table 1, the decrease was on the order of 0.02 to 0.03 ppm between successive monthly analyses. In others, it was much greater than 0.03 ppm/month.

The accuracy of the supplier's analyses improved in the Group 2 cylinders evaluated over a 12-month period. As seen in Table 2, these samples were unstable. The average loss of SO_2 in this group was 76%, or 0.03 ppm/month for the life of the survey.

With the exception of one sample, the vendor accuracy of the cylinders in Group 3 (Table 3) was on the same order as those of Group 2. This stability assessment began in May 1977 and continued through March 1978. The average rate of SO_2 loss was 0.006 ppm/month. Although not completely stable, this rate of decline was the lowest obtained up to that point, and represented a substantial improvement over the cylinders examined earlier.

With the acquisition of two additional cylinders in July 1977, another series of tests was begun. Four more aluminum and two stainless steel cylinders

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TABLE 1. SO_2 EVALUATION CYLINDERS: GROUP 1

Cylinder Number	Supplier Certification (ppm SO_2)	Analysis Date 1/76	2/76	3/76	4/76	5/76
FF 2249	1.40	0.89	0.87	0.85	0.79	0.69
FF 2124	1.30	0.70	0.72	0.67	0.64	0.61
FF 2136	1.00	-	0.57	-	0.52	0.45
FF 2244	1.00	-	0.50	0.44	0.41	0.45
FF 2246	1.40	0.76	0.77	0.70	0.68	0.37
FF 2245	0.50	0.29	0.32	0.29	0.26	0.23

TABLE 2. SO_2 EVALUATION CYLINDERS: GROUP 2

Cylinder Number	Supplier Certification (ppm SO_2)	Analysis Date 1/76	3/76	4/76	5/76	7/76	10/76	12/76
CC 127	0.33	0.32	0.29	0.24	0.20	0.15	0.11	0.08
CC 141	0.38	0.31	0.25	0.23	0.19	0.14	0.10	0.08
CC 130	0.33	0.31	0.25	0.23	0.18	0.16	0.06	0.04
CC 140	0.54	0.47	0.38	0.36	0.24	0.22	0.16	0.12
CC 286	0.51	0.47	0.40	0.35	0.29	0.24	0.14	0.11
CC 146	0.58	0.66	0.59	0.53	0.46	0.39	-	-

TABLE 3. SO_2 EVALUATION CYLINDERS: GROUP 3

Cylinder Number	Supplier Certification (ppm SO_2)	Analysis Date 5/77	7/77	12/77	1/78	3/78
CC 5100	0.45	0.44	0.46	0.34	0.38	-
MM 13672	0.16	0.07	0.05	0.05	0.04	0.04
CC 4750	0.13	0.11	0.10	0.07	0.08	-
CC 5092	0.31	0.30	0.30	-	0.25	0.24

TABLE 4. SO_2 EVALUATION CYLINDERS: GROUP 4

Cylinder Number	Supplier Certification (ppm SO_2)	Analysis Date 12/77	1/78	3/78
SS C118*	0.544	0.54	0.45	0.45
SS C105*	0.265	0.13	0.00	-
LL 4406	0.10	0.12	0.11	0.10
LL 4404	0.10	0.13	0.12	0.11
LL 4412	0.10	0.12	0.11	0.11
LL 4408	0.10	0.11	0.11	0.11
LL 4512	0.14	0.11	0.09	0.08

*Stainless steel cylinder.

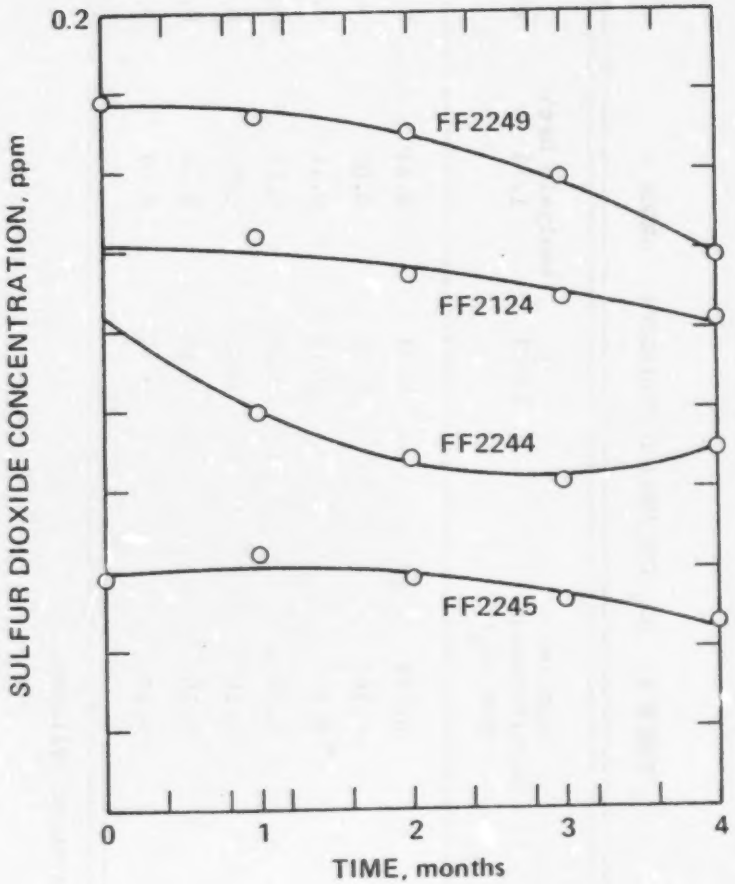

Figure 4. Stability of Group 1 SO_2 compressed gas samples.

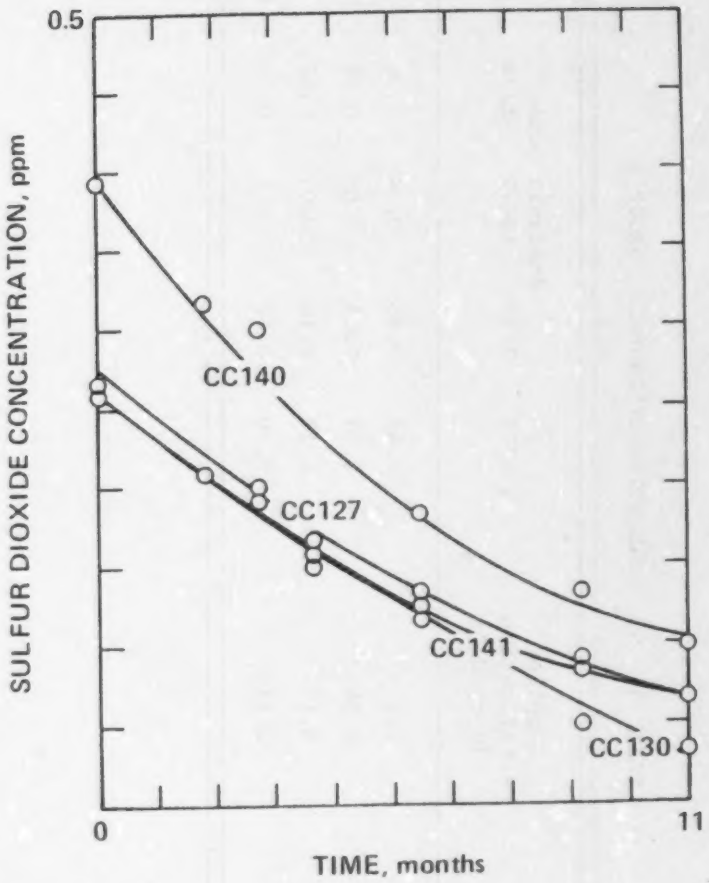

Figure 5. Stability of Group 2 SO_2 compressed gas samples.

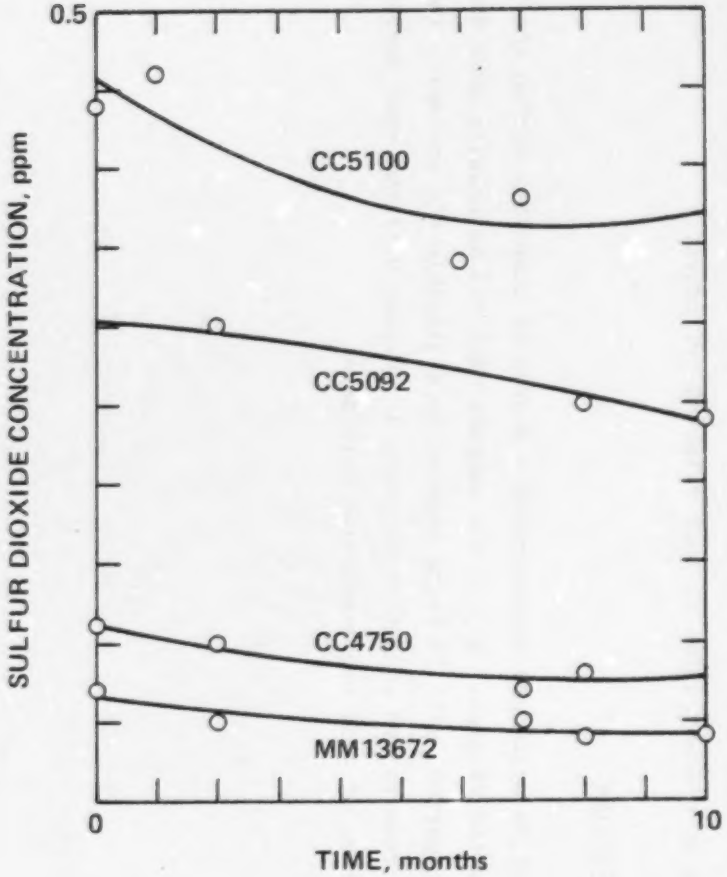

Figure 6. Stability of Group 3 SO_2 compressed gas samples.

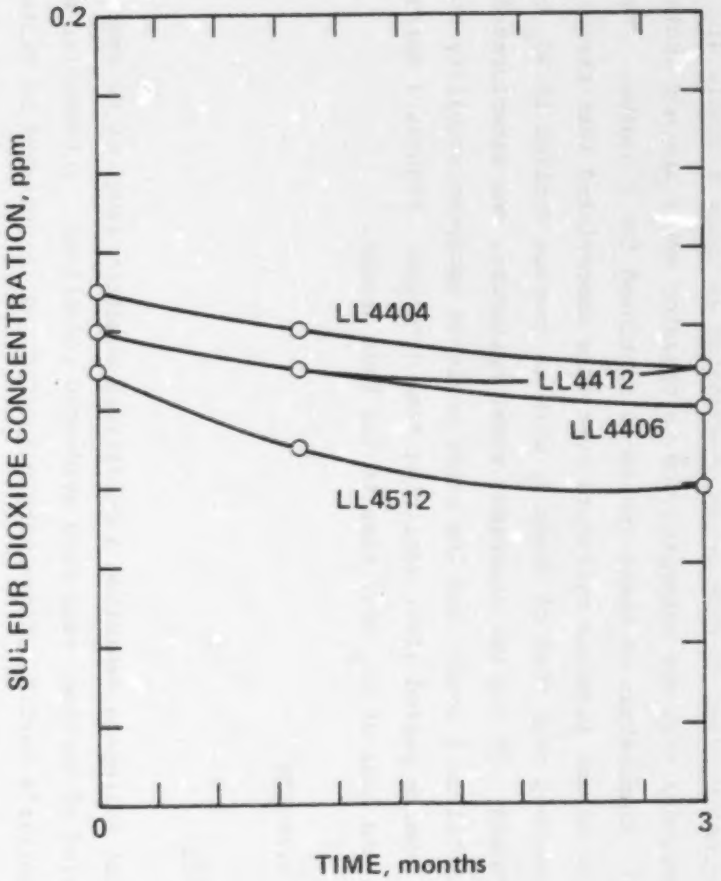

Figure 7. Stability of Group 4 SO_2 compressed gas samples.

were subsequently added to this group, designated as Group 4 (Table 4). (Data from one cylinder that was exhausted due to operator error are not included in the table.) Examination of these specimens continued for 3 months. The degree of stability of the aluminum cylinders over this abbreviated test period was somewhat improved over that of Group 3, with an average decline in SO_2 of 0.004 ppm/month. Of the two stainless steel cylinders, one deteriorated from 0.13 ppm to nil in 1 month, and the other achieved apparent stability at 0.45 ppm for a 2-month period after declining from 0.54 ppm. Figures 4 through 7 illustrate the loss of SO_2 over time in the four groups.

OXIDES OF NITROGEN

Nitric Oxide

Several cylinders exhibited a relatively constant level of NO over an extended period of testing; many more evidenced reductions. Substantial errors in the supplier's certifications were suspected, as illustrated in Tables 5 and 6. The single stainless steel cylinder admitted to the evaluation in November 1977 exhibited a consequential reduction in NO. Figure 8 shows the changes in NO concentration of four selected cylinders.

Nitrogen Dioxide

Ten of 26 cylinders demonstrated a degree of stability during the first 3 months after receipt. All of the samples declined materially over the 10-month test period, with 10 being reduced to negligible NO_2 content. The cumulative test results are presented in Table 7. Figure 9 illustrates decline in concentration of four representative cylinders.

TABLE 5. NO EVALUATION CYLINDERS

Cylinder Number	Supplier Certification (ppm NO)	Analysis Date 3/76	5/76	12/76	11/77	2/78
FF 1573	1.03	1.12	1.09	1.09	0.96	0.95
FF 1587	1.02	1.14	1.11	1.12	1.01	1.04
FF 1634	1.03	1.13	1.10	1.11	0.99	0.97
FF 1612	1.02	1.12	1.09	1.12	0.98	0.99
FF 1595	1.02	1.11	1.08	1.03	0.93	0.96
FF 1584	1.03	1.12	1.07	1.06	0.97	1.00
FF 1583	1.02	1.11	1.07	1.06	0.98	1.00
FF 1607	1.03	1.14	1.09	1.07	0.98	0.97
FF 1585	0.51	0.56	0.53	0.53	0.46	0.47
FF 1054	0.51	0.57	0.53	0.52	0.46	0.46
FF 1456	0.52	0.57	0.55	0.55	0.48	0.49
FF 1588	0.51	0.56	0.53	0.51	0.47	-
FF 2501	0.30	0.24	0.23	0.20	0.24	0.23
FF 2474	0.30	0.25	0.21	0.17	0.18	-
SSC 122*	0.44	-	-	-	0.38	0.35

*Stainless steel cylinder.

TABLE 6. NO EVALUATION CYLINDERS

Cylinder Number	Supplier Certification (ppm NO)	Analysis 3/76	5/76	12/76
FF 1592	1.02	1.13	1.09	1.11
FF 2504	0.31	0.26	0.24	0.25
FF 2505	0.30	0.24	0.21	0.21
FF 2486	0.29	0.24	0.20	0.21
FF 2806	0.30	0.25	0.23	0.23
FF 1635	0.52	0.56	0.54	0.52
FF 1060	0.51	0.55	0.53	0.50
FF 1598	0.51	0.56	0.52	0.53
FF 2810	0.30	0.25	0.22	0.21
FF 2490	0.31	0.25	-	0.25
FF 2506	0.30	0.25	-	0.21

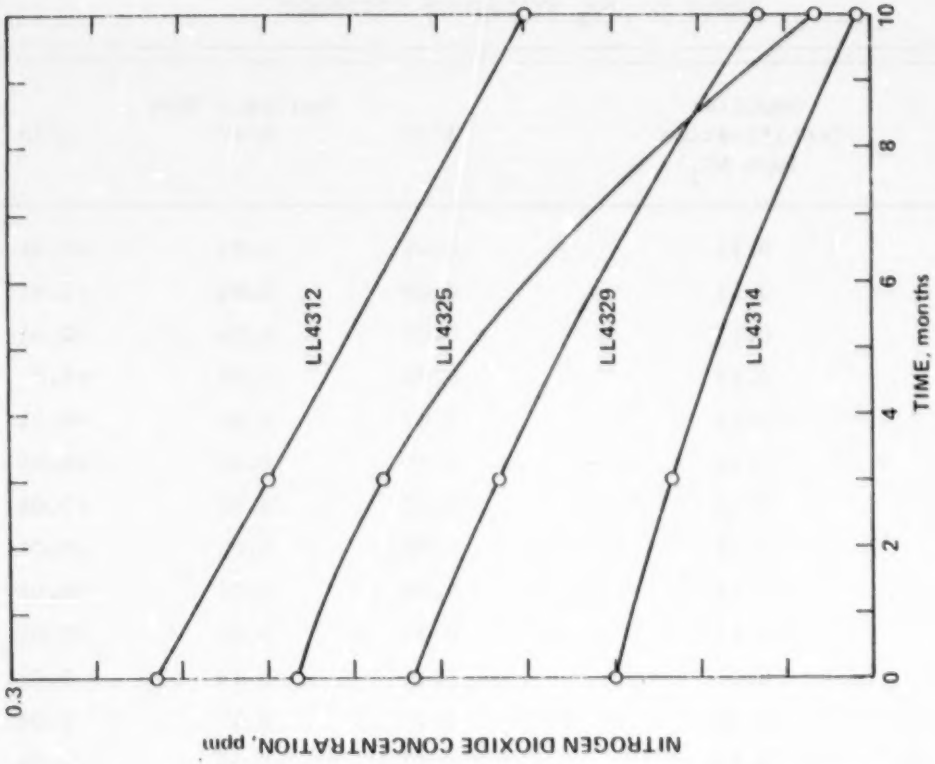

Figure 9. Stability of NO_2 compressed gas samples.

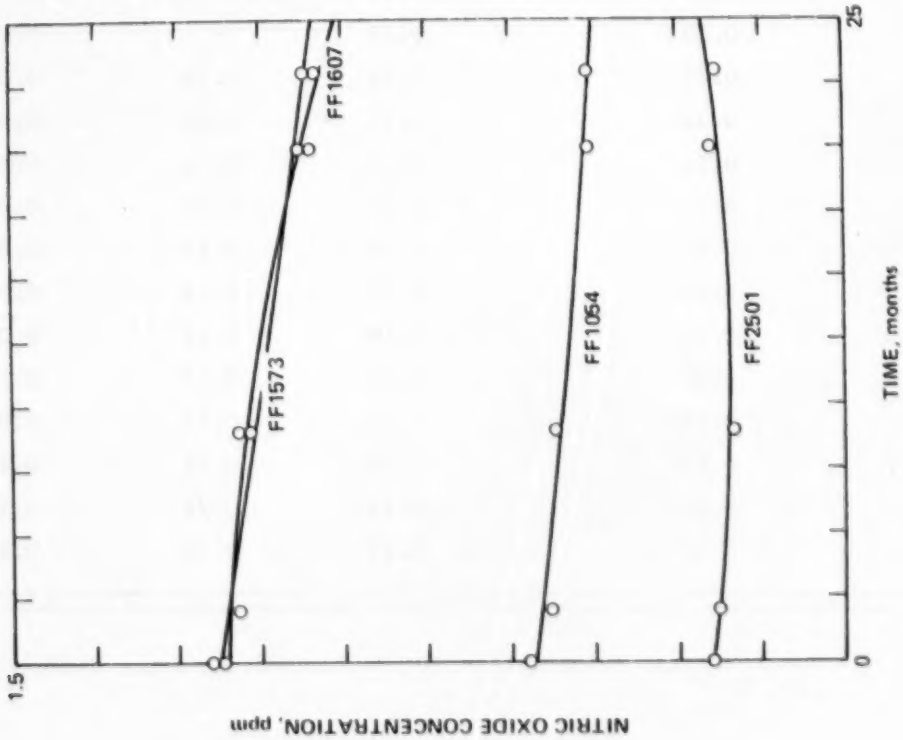

Figure 8. Stability of NO compressed gas samples.

TABLE 7. NO_2 EVALUATION CYLINDERS

Cylinder Number	Supplier Certification (ppm NO_2)	Analysis Date 3/77	6/77	1/78
LL 4315	0.11	0.05	0.03	<0.01
LL 4331	0.13	0.08	0.06	<0.01
LL 4310	0.13	0.08	0.08	<0.01
LL 4354	0.13	0.08	0.07	<0.01
LL 4338	0.12	0.07	0.06	<0.01
LL 4321	0.11	0.06	0.05	<0.01
LL 4324	0.12	0.07	0.05	<0.01
LL 4327	0.14	0.08	0.08	<0.01
LL 4314	0.13	0.09	0.07	<0.01
LL 4333	0.12	0.07	0.06	<0.01
LL 4313	0.23	0.16	0.14	0.05
LL 4329	0.20	0.16	0.13	0.04
LL 4358	0.23	0.17	0.15	0.06
LL 4320	0.23	0.19	-	0.11
LL 4359	0.22	0.16	0.14	0.06
LL 4353	0.24	0.17	0.16	0.08
LL 4339	0.22	0.14	0.11	0.02
LL 4349	0.22	0.17	0.16	0.08
LL 4346	0.27	0.19	0.17	0.06
LL 4335	0.26	0.17	0.14	0.05
LL 4326	0.27	0.18	0.14	0.02
LL 4325	0.28	0.20	0.17	0.02
LL 4318	0.28	0.17	0.15	0.08
LL 4348	0.32	0.22	0.21	0.12
LL 4328	0.32	0.23	0.24	0.16
LL 4312	0.32	0.25	0.21	0.12

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16. ABSTRACT

Compressed gas samples of sub-part per million concentrations of sulfur dioxide, nitric oxide, and nitrogen dioxide were evaluated for long and short term stability. Except for several stainless steel tanks, the samples were contained in aluminum cylinders. A degree of stability was achieved over the short term, however, all of the samples were considered unstable over the term of the evaluations. Utilization of ambient level compressed gas samples may be considered where relative stability is required for no more than two or three months.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Air Pollution Gas Analysis	SO_2 NO_2^2 Stability Cylinders	13B 14B

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